PATENT

Docket No. A-5720

Box Patent Application
Commissioner of Patents and Trademarks
Washington, D.C. 20231

NEW APPLICATION TRANSMITTAL

Transmitted herewith for filing is the patent application of

Inventor(s): **Jürgen Schulz-Harder** and **Karsten Schmidt**

WARNING: *Patent must be applied for in the name(s) of all of the actual inventor(s). 37 CFR 1.41(a) and 1.53(b).*

For (title): **PROCESS FOR PRODUCING A CERAMIC SUBSTRATE AND A CERAMIC SUBSTRATE**

1. Type of Application

This new application is for a(n) *(check one applicable item below):*

[X] Original

[ ] Design

[ ] Plant

WARNING: *Do not use this transmittal for a completion in the U.S. of an International Application under 35 U.S.C. 371(c)(4) unless the International Application is being filed as a divisional, continuation or continuation-in-part application.*

NOTE: If one of the following 3 items apply, then complete and attach ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF A PRIOR U.S. APPLICATION CLAIMED.

[ ] Divisional

[ ] Continuation

[ ] Continuation-in-part (CIP)

CERTIFICATION UNDER 37 CFR 1.10

I hereby certify that this New Application Transmittal and the documents referred to as enclosed therein are being deposited with the United States Postal Service on this date____________ in an envelope as "Express Mail Post Office to Addressee" Mailing Label Number__________ addressed to the: Commissioner of Patents and Trademarks, Washington, D.C. 20231.

____
(Type or print name of person mailing paper)

____
(Signature of person mailing paper)

NOTE: Each paper or fee referred to as enclosed herein has the number of the "Express Mail" mailing label placed thereon prior to mailing 37 CFR 1.10(b).

2. Benefit of Prior U.S. Application(s) (35 USC 120)

NOTE: If the new application being transmitted is a divisional, continuation or a continuation-in-part of a parent case, or where the parent case is an International Application which designated the U.S., then check the following item and complete and attach ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OR PRIOR U.S. APPLICATION(S) CLAIMED.

[ ] The new application being transmitted claims the benefit of prior U.S. application(s) and enclosed are ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED.

3. Papers Enclosed which are Required for Filing Date under 37 CFR 1.53(b)(Regular) or 37 CFR 1.153(Design) Application

12 Pages of Specification

3 Pages of Claims

1 Pages of Abstract

2 Sheets of Drawing

[ ] Formal

[X] Informal

WARNING: DO NOT submit original drawings. A high quality copy of the drawings should be supplied when filing a patent application. The drawings that are submitted to the Office must be on strong, white, smooth, and non-shiny paper and meet the standards according to § 1.84. If corrections to the drawings are necessary, they should be made to the original drawing and a high-quality copy of the corrected original drawing then submitted to the Office. Only one copy is required or desired. Comments on proposed new 37 CFR 1.86. Notice of March 9, 1988 (1990 O.G. 57-62).

NOTE: "Identifying indicia such as the serial number, group art unit, title of invention, attorney's docket number, inventor's name, number of sheets, etc., not to exceed 2¾ (7.0 cm) in width may be placed in a centered location between the side edges within three fourths inch (19.1 mm) of the top edge. Either this marking technique on the front of the drawing or the placement, although not preferred, of this information and the title of the invention on the back of the drawings is acceptable". Proposed 37 CFR 1.84(1). Notice of March 9, 1988 (1990 O.G. 57-62).

4. Additional Papers Enclosed

[X]Preliminary Amendment

[ ] Information Disclosure Statement

[ ] Form PTO-1449

[ ] Citations

[ ] Declaration of Biological Deposit

[ ] Submission of "Sequence Listing," computer readable copy and/or amendment pertaining thereto for biotechnology invention containing nucleotide and/or amino acid sequence.

[ ] Authorization of Attorney(s) to Accept and Follow Instructions from Representative.

[ ] Special Comments

[X]Other - Verified Translations, Marked-up Copy of Specification

5. **Declaration or Oath**

[ ] Enclosed
executed by *(check all applicable boxes)*

[ ] inventor(s)

[ ] legal representative of inventor(s). 37 CFR 1.42 or 1.43

[ ] joint inventor or person showing a proprietary interest on behalf of inventor who refused to sign or cannot be reached.

[ ] this is the petition required by 37 CFR 1.47 and the statement required by 37 CFR 1.47 is also attached. *See Item 13 below for fee.*

[X] Not Enclosed.

WARNING: *Where the filing is a completion in the U.S. of an International Application but where a declaration is now available or where the completion of the U.S. application contains subject matter in addition to the International Application, the application may be treated as a continuation or continuation-in-part, as the case may be, utilizing ADDED PAGE FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION CLAIMED.*

[ ] Application is made by a person authorized under 37 CFR 1.41(c) on behalf of *all* the inventor(s). The declaration or oath, along with the surcharge required by 37 CFR 1.16(e) can be filed subsequently.

NOTE: *It is important that all the correct inventor(s) are named for filing under 37 CFR 1.41(c) and 1.53(b).*

[ ] Showing that the filing is authorized. (*Not required unless called into question. 37 CFR 1.41(d)*).

6. **Inventorship Statement**

WARNING: *If the named inventors are each not the inventors of all the claims, an explanation, including the ownership of the various claims at the time the last claimed invention was made, should be submitted.*

The inventorship for all the claims in this application are:

[X]The same

or

[ ] Are not the same. An explanation, including the ownership of the various claims at the time the last claimed invention was made,

[ ] is submitted.

[ ] will be submitted.

7. **Language**

NOTE: *An application including a signed oath or declaration may be filed in a language other than English. A verified English translation of the non-English language application and the processing fee of $130.00 required by 37 CFR 1.17(k) is required to be filed with the application or within such time as may be set by the Office. 37 CFR 1.52(d).*

NOTE: *A non-English oath or declaration in the form provided or approved by the PTO need not be translated. 37 CFR 1.69(b).*

[X] English

[ ] non-English

[X] the attached translation is a verified translation. 37 CFR 1.52(d).

8. **Assignment**

[ ] An assignment of the invention to ______________________________

____

____

[ ] is attached. (A separate "ASSIGNMENT COVER LETTER ACCOMPANYING NEW PATENT APPLICATION" is also attached.

[ ] will follow.

NOTE: *"If an assignment is submitted with a new application, send two separate letters-one for the application and one for the assignment." Notice of May 4, 1990 (1114 O.G. 77-78).*

9. **Certified Copy**

Certified copy(ies) of application(s)

Germany	196 03 822.7-42	02-02-96
(country)	(appln. no.)	(filed)
(country)	(appln. no.)	(filed)
(country)	(appln. no.)	(filed)

from which priority is claimed.

[ ] is (are) attached.

[X] will follow.

NOTE: *The foreign application forming the basis for the claim for priority must be referred to in the oath or declaration. 37 CFR 1.55(a) and 1.63.*

NOTE: *This item is for any foreign priority for which the application being filed directly relates. If any parent U.S. application or International Application from which this application claims benefit under 35 U.S.C. 120 is itself entitled to priority from a prior foreign application then complete item 18 on the ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED.*

10. **Fee Calculation (37 CFR 1.16)**

A. **[X] Regular application**

CLAIMS AS FILED				
Number Filed		**Number Extra**	**Rate**	**Basic Fee** $ 770.00
Total Claims 37 CFR 1.16(c)	18 - 20	= __	x $ 22.00	= $
Independent Claims 37 CFR 1.16(b)	2 - 3	= __	x $ 80.00	= $
Multiple Dependent Claim(s), if any 37 CFR 1.16(d)				= $ 260.00

[ ] Amendment cancelling extra claims enclosed.

[ ] Amendment deleting multiple-dependencies enclosed.

[ ] Fee for extra claims is not being paid at this time.

NOTE: *If the fees for extra claims are not paid on filing, they must be paid or the claims cancelled by amendment, prior to the expiration of the time period set for response by the Patent and Trademark Office in any notice of fee deficiency. 37 CFR 1.16(d).*

Filing Fee Calculation $ 770.00

B. [ ] **Design Application**
($310.00-37 CFR 1.16(f))

Filing Fee Calculation $____________________

C. [ ] **Plant Application**
($510.00-37 CFR 1.16(g))

Filing Fee Calculation $____________________

11. Small Entity Statement(s)

[ ] Verified Statement(s) that this is a filing by a small entity under 37 CFR 1.9 and 1.27 is(are) attached.

Filing Fee Calculation (50% of A, B or C above) $____________________

NOTE: *Any excess of the full fee paid will be refunded if a verified statement and a refund request are filed within 2 months of the date of timely payment of a full fee. 37 CFR 1.26(a).*

12. Request for International-Type Search (37 CFR 1.104(d)) *(complete, if applicable)*

[ ] Please prepare an international-type search report for this application at the time when national examination on the merits takes place.

13. Fee Payment Being Made at this Time

[X] Not Enclosed

[X] No filing fee is to be paid at this time. *(This and the surcharge required by 37 CFR 1.16(e) can be paid subsequently.)*

[ ] Enclosed

[ ] Basic Filing Fee $____________________

[ ] Recording Assignment ($40 - 37 CFR 1.21(h)) $____________________

[ ] Petition fee for filing by other than all the inventors or person on behalf of the inventor where inventor refused to sign or cannot be reached. ($120.00 - 37 CFR 1.47 and 1.17(h)) $____________________

[ ] For processing an application with a specification in a non-English language. ($130 - 37 CFR 1.52(d) and 1.17(k)) $____________________

[ ] Processing and retention fee ($130 - 37 CFR 1.53(d) and 1.21(l)) $____________________

[ ] Fee for international-type search report (37 CFR 1.21(e)) $____________________

NOTE: *37 CFR 1.21(f) establishes a fee for processing and retaining any application which is abandoned for failing to complete the application pursuant to 37 CFR 1.53(d) and this, as well as the changes to 37 CFR 1.53 and 1.78, indicate that in order to obtain the benefit of a prior U.S. application, either the basic filing fee must be paid or the processing and retention fee of § 1.21(l) must be paid within 1 year from notification under § 53(d).*

Total Fees Enclosed $____________________

14. Method of Payment of Fees

[ ] Check in the amount of $________

[ ] Charge Account No. 08-2455 in the amount of $__________. A duplicate copy of this transmittal is attached.

NOTE: *Fees should be itemized in such a manner that it is clear for which purpose the fees are paid. 37 CFR 1.22(b).*

15. Authorization to Charge Additional Fees

WARNING: *If no fees are to be paid on filing, the following items should not be completed.*

WARNING: *Accurately count claims, especially multiple dependent claims, to avoid unexpected high charges, if extra claim charges are authorized.*

[ ] The Commissioner is hereby authorized to charge the following additional fees by this paper and during the entire pendency of this application to Account No. 08-2455.

[ ] 37 CFR 1.16(a), (f) or (g) (filing fees)

[ ] 37 CFR 1.16(b), (c) and (d) (presentation of extra claims)

NOTE: *Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must only be paid or these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 CFR 1.16(d)), it might be best not to authorize the PTO to charge additional claim fees, except possibly when dealing with amendment after final action.*

[ ] 37 CFR 1.16(e) (surcharge for filing the basic filing fee and/or declaration on a date later than the filing date of the application)

[ ] 37 CFR 1.17 (application processing fees)

WARNING: *While 37 CFR 1.17(a), (b), (c) and (d) deal with extensions of time under § 1.136(a), this authorization should be made only with the knowledge that: "Submission of the appropriate extension fee under 37 CFR 1.136(a) is to no avail unless a request or petition for extension is filed." (Emphasis added). Notice of November 5, 1985 (O.G. 27).*

[ ] 37 CFR 1.18 (issue fee at or before mailing of Notice of Allowance, pursuant to 37 CFR 1.311(b))

NOTE: *Where an authorization to charge the issue fee to a deposit account has been filed before the mailing of a Notice of Allowance, the issue fee will be automatically charged to the deposit account at the time of mailing the Notice of Allowance. 37 CFR 1.311(b).*

NOTE: *37 CFR 1.28(b) requires "Notification of any change in loss of entitlement to small entity status must be filed in the application...prior to paying, or at the time paying...Issue Fee". From the wording of 37 CFR 1.26(b): (a) notification of change of status must be made even if the fee is paid as "other than a small entity" and (b) no notification is required if the change is to another small entity.*

16. Instructions as to Overpayment

[X]Credit Account No. 08-2455.

[ ] Refund

Stewart L. Gitler

SIGNATURE OF ATTORNEY

Stewart L. Gitler

Type or print attorney name

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[ ] **Incorporation by reference of added pages**

Check the following item if the application in this transmittal claims the benefit of prior U.S. application(s) (including an international application entering the U.S. stage as a continuation, divisional or C-I-P application) and complete and attach the ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED

[ ] Plus Added Pages for New Application Transmittal Where Benefit of Prior U.S. Application(s) Claimed

Number of pages added____________________

[ ] Plus Added Pages For Papers Referred To In Item 4 Above

Number of pages added____________________

[ ] Plus "Assignment Cover Letter Accompanying New Application"

Number of pages added____________________

[ ] **Statement Where No Further Pages Added**

(If no further pages form a part of this Transmittal then end this Transmittal with this page and check the following item)

[X]This transmittal ends with this page.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Jürgen Schulz-Harder et al

Group Art Unit:

Serial No:

Examiner:

Filed :

For : **PROCESS FOR PRODUCING A CERAMIC SUBSTRATE AND A CERAMIC SUBSTRATE**

PRELIMINARY AMENDMENT

Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

Please amend the above-identified application as follows:

IN THE SPECIFICATION:

Please replace the original specification with the attached "Substitute Specification."

IN THE CLAIMS:

Please delete "Claims," and insert in its place --**WHAT IS CLAIMED IS**:--.

Please cancel claims 1-17, and replace same with new claims 18-35.

--18. A process for producing a substrate with at least one layer of an aluminum nitrate ceramic, comprising the steps of applying an auxiliary or intermediate layer of an aluminum oxide to at least one side of said at least one layer, wherein said auxiliary intermediate layer is applied using a copper oxide-containing material such that said auxiliary or intermediate layer, such that a part of a thickness of said auxiliary or intermediate layer contains about 0.05-44 percent

by weight of a copper oxide and said at least one layer of aluminum nitride ceramic of said at least one side is provided with a layer of copper or copper oxide or other copper-containing compounds having a thickness of about 1.5×10^{-4} micron thick to form a substrate; and then treating said substrate at a temperature ranging from about 800 - 1300°C is in an oxygen-containing atmosphere until said intermediate layer with a desired thickness has formed.

19. A process according to claim 18, wherein treatment in said oxygen-containing atmosphere lasts until a layer thickness of about 0.5 - 10 microns has developed for said intermediate layer.

20. A process according to claim 18, wherein said aluminum nitride ceramic is treated in an oxygen-containing atmosphere, at the same time said copper-oxide containing material is reacted via the gaseous phase with said aluminum oxide.

21. A process according to claim 20, wherein said treatment in said oxygen-containing atmosphere with a copper-oxide-containing material lasts until a layer thickness of roughly 0.5 - 10 microns has developed for said intermediate layer.

22. A process according to claim 18, wherein after producing at least one intermediate layer, a metal layer is attached to said at least one intermediate layer over its surface using an oxidized metal or copper foil and further using a DCB process.

23. A process according to claim 18, wherein said at least one layer of aluminum nitride ceramic is provided on both sides with said auxiliary or intermediate layer, and wherein one metal or copper layer is applied to each of said intermediate layers using a DCB process.

24. A substrate comprising at least one layer of an aluminum nitride ceramic, an intermediate or auxiliary layer which comprises aluminum oxide (Al_2O_3) and which has a thickness of about roughly 0.5 - 10 microns provided on said surface of said at least one layer of an aluminum nitride ceramic, wherein said intermediate layer contains 0.05-44 percent by weight of at least one copper oxide, and wherein a portion of copper oxide in said intermediate layer is uniformly distributed in clusters.

25. A substrate according to claim 24, wherein said clusters have a diameter of less than about 0.01 microns.

26. A substrate according to claim 24, wherein said intermediate layer comprises a first layer of aluminum oxide which is directly adjacent to said layer of aluminum nitride ceramic and a copper oxide, and a second aluminum oxide layer which contains no copper or copper oxide.

27. A substrate according to claim 24, wherein a concentration of said copper oxide in said intermediate layer decreases with an increasing distance from said layer of aluminum nitride ceramic.

28. A substrate according to claim 24, wherein said portion of copper oxide present in said aluminum oxide is copper-aluminum-spinel ($CuAl_2O_4$ or CuAlO).

29. A substrate according to claim 24, wherein said at least one layer of aluminum nitride is joined flat on one side via said intermediate layer with a 0.1 - 0.8 mm thick copper layer or metal coating.

30. A substrate according to claim 24, wherein said copper-oxide layer is joined via an oxygen-rich copper phase, having a thickness of at least three microns, to one side of said at least on layer of aluminum nitride ceramic or to said intermediate layer.

31. A substrate according to claim 24, wherein said at least one layer of aluminum nitride contains about 0.5 - 6 percent by weight of a binder.

32. A substrate according to claim 31, wherein said binder is CaO.

33. A substrate according to claim 24, wherein a binder is present in said intermediate layer.

34. A substrate according to claim 33, wherein said binder is CaO.

35. A substrate according to claim 24, wherein at least one copper layer is applied to at least one side of said at least one layer of aluminum nitride ceramic or to said intermediate layer in a DCB process.--

REMARKS

Applicant has replaced the original specification with a substitute specification. Applicant includes a marked-up copy of the original specification for the examiner to compare. No new matter has been inserted into the specification in the substitute specification and only changes to conform the specification with U.S. practice have been made.

Applicant has cancelled claims 1-17 and replaced same with claims 18-35 to also place same in a better condition for review and examination. Multiple dependencies and a number of antecedent basis problems have been addressed.

Please feel fee to contact the undersigned with questions.

Respectfully submitted,

by _Stewart L. Gitler_
Stewart L. Gitler, Reg. 31,256

February 3, 1997

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Attorney's Docket: A-4720.PAM/lat

Marked Up Copy of Specification

Process for producing a ceramic substrate and a ceramic substrate

BACKGROUND OF THE INVENTION

The invention relates to a process for producing a substrate with at least one layer of aluminum nitride ceramic, in which ~~process~~ one auxiliary or intermediate layer of aluminum oxide is applied to at least one side of this layer.

The invention further relates ~~furthermore~~ to a substrate with at least one layer ~~layer~~ ~~essentially~~ of aluminum nitride (AlN) which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide (Al_2O_3) and which has a thickness in the range of roughly 0.5 - 10 microns.

Ceramic substrates for electrical circuits or modules, especially for power circuits or modules, are known in the most varied versions. In particular, it is known to produce a ~~producing the~~ metal coating, required for making printed conductors, terminals, etc., on an aluminum oxide ceramic using the so-called "DCB process" (direct copper bond technology) and using copper foils which are oxidized on their surfaces and which form the metallic coating. ~~is known.~~ The copper oxide layer of these foils forms a eutectic with a melting point below the melting point of the copper so that by placing the foils on the ceramic and by heating all the layers they can be joined to one another, especially by melting on the copper, ~~essentially~~ only in the area of the oxide layer. The DCB process is a technique known to one skilled in the art.

~~Especially~~ In power circuits, ~~is~~ the use of an aluminum nitride ceramic instead of an aluminum oxide ceramic is desirable

due to the thermal conductivity of the aluminum nitride ceramic, which is higher than that of the aluminum oxide ceramic.

In this case, ~~But here~~ ~~the disadvantage is essentially that the conventional and proven~~ the DCB process cannot be easily used for ~~in~~ aluminum nitride ceramics.

It ~~is an objective~~ has been suggested to ~~has therefore be suggested that~~ first apply a layer of aluminum oxide ~~be applied~~ to the layer of aluminum nitride ceramic, such that this intermediate or auxiliary layer ~~which is designed to~~ enables the application of a metallic coating or the copper layer using the DCB process. This intermediate layer has a roughness which improves adhesion on its exposed surface (DE-OS 35 34 886).

Furthermore, it was proposed that an aluminum oxide layer be applied to an initial substrate or carrier body of aluminum nitride, ~~especially~~ by flame spraying an aluminum oxide powder or in a screen printing process (DE-OS 38 44 264).

In all these known DCB processes, the disadvantage is that in spite of applying the auxiliary or intermediate layer of aluminum oxide, a flawless homogenous flat bond without faults is not achieved between the layer of aluminum nitride ceramic and the metal coating ~~is not achieved in the DCB process~~, but rather numerous faults occur, i.e., areas in which no bond has been formed or the metal coating has been lifted away from the ceramic by bubble formation. Thus the adhesive strength of the metal coatings and the thermal conductivity of the substrates overall are adversely affected.

To improve adhesive strength or bond quality, i.e., to reduce the bubble portion, it is proposed in DE 41 04 860.1 that oxidation of the aluminum nitride (AlN) be done to form the

intermediate layer of aluminum oxide (Al_2O_3). This is accomplished in an oxygen atmosphere free of water vapor.

Conversely, it is proposed in WO 92/11 113 that the aluminum nitride be oxidized and cooled in a controlled manner under a water vapor-containing atmosphere to form the intermediate layer of aluminum oxide.

In JP 02-124 773 A (in Patent Abstracts of Japan C - 743, Vol. 14/No. 342) it is proposed that the adhesive strength of the metal coating be increased by incorporating CaO and SiO_2 into the aluminum oxide (Al_2O_3) layer, CaO and SiO_2 originating from the sintering aids of the aluminum nitride (AlN) body.

One disadvantage of the aforementioned ~~known~~ processes, ~~is among others~~ is that the process parameters must be very accurately maintained and further these processes are not applicable ~~be applied especially~~ to highly heat-conductive aluminum nitride which contains yttrium oxide (Y_2O_3) as the sintering aid.

In JP 03-228 885 A (in Patent Abstracts of Japan C-890, Vol. 16/No. 1) it is proposed in order to increase the adhesive strength that the aluminum oxide layers be doped with one or more elements of the group Ti, V, Mo, Mb, W, Co or Ni.

In WO 92/11 113 reference is made to a publication of Kuromitsu which relates to SiO_2-Al_2O_3 intermediate layers for increasing adhesive strength.

In our own extensive studies, however, the described results of the aforementioned known processes could not be confirmed. It is thus apparent that the process parameters must be precisely

controlled ~~such that~~ or negative results occur even with small deviations.

The incorporation of SiO_2 cited in WO 92/11 113 moreover also engenders the danger that SiO_2 is present not as SiO_2 bound as mullite, but as free SiO_2 which reacts at the eutectic temperature with copper oxide to form a liquid phase and consumes the Cu-Cu_2O eutectic which is necessary for the DCB process, with which the adhesive strength or bond quality would be strongly reduced.

The object of the invention is to devise a process for producing a ceramic substrate which avoids the aforementioned disadvantages and makes it possible to apply the metal coating to an aluminum oxide ceramic over a large area without faults.

SUMMARY OF THE INVENTION

To achieve this object a process for producing a substrate with at least one layer of aluminum nitride ceramic, in which ~~(process)~~ an auxiliary or intermediate layer of aluminum oxide is applied to at least one side of this layer is characterized in that the intermediate layer is applied using a copper oxide-containing material such that the intermediate layer at least in part of its thickness contains a portion of copper oxide of roughly 0.05-44 percent by weight and that the layer of aluminum nitride first on at least one surface side is provided with a roughly 1.5×10^{-4} to 1200×10^{-4} micron thick layer of copper or copper oxide or other copper-containing compounds and then at a temperature between roughly 800 - 1300°C is treated in an oxygen-containing atmosphere until an intermediate layer with the desired thickness ~~as~~ is formed.

A substrate is formed with at least one layer which comprises ~~consists essentially of~~ aluminum nitride (AlN) and which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide (Al_2O_3) and has a thickness in the range from roughly 0.5 to 10 microns, and is characterized in that the intermediate layer has an ~~as the~~ additive present from ~~contains~~ 0.05-44 percent by weight of at least one copper oxide and that the proportion of copper oxide in the intermediate layer is ~~provided~~ uniformly distributed in clusters.

In the invention the intermediate layer applied to the aluminum nitride layer contains a small portion of copper oxide which is present and bound in the form of a spinel, for example, as $CuAl_2O_4$. Surprisingly, considerable compaction of the intermediate or auxiliary layer of aluminum oxide occurs due to the portion of copper oxide, so that diffusion of the nitrogen through the intermediate layer which is responsible for the faults and bubble formation in the prior art is effectively prevented. According to one finding underlying the invention, the improved seal can be attributed to the fact that by adding copper oxide in the intermediate or auxiliary layer those temperature changes which a substrate inevitably undergoes in ~~the~~ conventional processes cannot lead to cracks or ~~similar~~ leaks in this intermediate layer. In this way, the passage of gas through this layer ~~and thus especially~~ and as in the DCB process, ~~also~~ the reaction of the aluminum nitride with the oxygen or the copper oxide of the copper foils, ~~used in the DCB process~~ which normally causes the bubble fault, is ~~are~~ effectively prevented. Compared to ~~the~~ a pure Al_2O_3

layer, ~~in addition in the invention~~ the reactivity of ~~the~~ an Al_2O_3-$CuAl_2O_4$-intermediate layer with the liquid Cu-Cu_2O eutectic, is greatly improved. ~~during the DCB process~~.

According to the finding underlying the invention the improved seal can be attributed to the reduction of pore volume and prevention of crack formation during the temperature changes which necessarily occur and which a substrate undergoes in the conventional processes as a result of the incorporation of copper oxide into the Al_2O_3 phase.

BRIEF DESCRIPTION OF THE DRAWINGS

~~Developments of the invention are the subject of the subclaims~~.

over

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The invention is detailed below using Figures 1-5 which each reproduce in a simplified representation and in cross section various possible embodiments of the substrate according to the invention.

In the figures, the initial substrate or a layer of aluminum nitride ceramic (AlN) is labelled 1.

In the embodiment assumed for Figure 1 this initial substrate 1 is to be ~~provided~~ produced, using the DCB process known to one skilled in the art, with a metal coating on each of the two sides, i.e., with a thin copper layer or foil 2, each copper foil 2 being oxidized on both surface sides, i.e., consisting of core 3 of copper (Cu) and thin copper oxide layer 4 (Cu_2O/CuO) on both surface sides. Core 3 has a much greater thickness than that of two oxide layers 4.

To be able to bond copper layers 2 using the DCB process to initial substrate 1, the latter in the embodiment shown in Figure

1 has in addition to core 5 of aluminum nitride ceramic (AlN) on both surface sides, layer 6 which consists of aluminum oxide (Al_2O_3) and which contains a portion of copper oxide (Cu_2O/CuO) in an amount between 0.05 to 44 percent by weight. The thickness of layers 6 is much less than the thickness of core 5 and is roughly between 0.5 and 10 microns.

It has been shown that only by means of the ~~portion~~ addition of copper oxide in the intermediate layer 6, is there a homogenous bond of initial substrate 1 with copper layers 2 ~~possible~~ produced even using the DCB process, i.e., ~~especially~~ a blanket bond without defects, i.e., without areas on which the bond between respective copper layer 2 and initial substrate 1 has not occurred or is disrupted by bubble formation.

As was detailed above, according to the finding underlying the invention this can only be achieved when intermediate layers 6 of aluminum oxide ceramic (Al_2O_3) which are fundamentally necessary for execution of the DCB process have the above described proportion of copper oxide, since in the conventional process steps major temperature changes of the initial substrate cannot be avoided ~~in particular~~ after applying intermediate layers 6, ~~and during the DCB process~~. These temperature fluctuations, according to the finding underlying the invention, in the absence of copper oxide in intermediate layers 6, lead ~~there~~ to cracks or leaks with the result that in the area of these leaks the aluminum nitride of core 5 reacts directly with the copper oxide of oxide layers 4, ~~without~~ preventing the desired bond from occurring between initial substrate 1 and the copper layers, ~~rather~~ and

nitrogen is released ~~with th~~e result ing in ~~of~~ bubble formation between respective copper layer 2 and the initial substrate, according to the following reaction equation:

$2AlN + 3Cu_2O$ ----- $Al_2O_3 + Cu + N_2$

Surprisingly, the proportion of copper oxide incorporated into intermediate layers 6 causes their compaction such that these leaks or cracks do not occur for the temperature differences which are inevitable in the process in intermediate layers 6, nor do the associated disadvantages arise.

Since the thickness of intermediate layers 6 is roughly between 0.5 and 10 microns, these intermediate layers are sufficient for reliable bonding of the initial substrate with copper layers 2; the special advantage of the aluminum nitride, specifically the thermal conductivity which is much improved compared to the aluminum oxide ceramic, is however not adversely affected overall.

The initial substrate can be produced for example such that on core 5 of the aluminum nitride (AlN), there is provided on each side, a very thin layer, i.e., a layer with a thickness of roughly 1.5×10^{-4} to 1200~~0~~ $\times 10^{-4}$ microns of copper or copper oxide or the corresponding ions of other copper-containing compounds ~~is applied~~ and afterwards this core 5 is treated at a temperature in the range between roughly 800-1300°C, preferable at a temperature of 1200°C, in an oxidizing atmosphere.

The following processes are suitable, for example, for producing the initial substrate:

Example 1

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the substrate by plasma etching;

2. Application of a uniform copper layer by sputtering onto both sides of the cleaned substrate with a total amount of 1.3 mg copper at a time.

3. Heating of the substrate in an atmosphere containing N_2 and O_2 in a proportion of 20:80 to a temperature of roughly 1280°C;

4. Keeping the substrate at a temperature of 1280°C for 30 minutes;

5. Cooling to room temperature;

6. Oxidizing a copper sheet measuring 100 x 100 mm and a thickness of 0.3 mm such that a uniform copper oxide layer of roughly 0.8 microns thick results;

7. Placing the copper sheet on the AlN substrate treated in steps 1-5;

8. Heating of the bond of substrate and copper sheet to 1071°C in a gas atmosphere consisting of nitrogen with an oxygen portion of 40 x 10^{-6};

9. Keeping the bond at the temperature for 2.5 minutes;

10. Cooling to room temperature.

A substrate of AlN with an intermediate layer of Al_2O_3 and $CuAl_2O_4$ and a securely bonded copper layer with a peel strength greater than 50 N/mm is obtained.

Example 2:

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the aluminum substrate with ultrasound in deionized water;

2. Currentless deposition of copper with a commercial bath until 1.8 mg copper total has been deposited on each side of the substrate;

3. This is followed by process steps 3-10 of example 1.

Example 3:

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the aluminum substrate with ultrasound in deionized water;

2. Heating of the substrate in a tube furnace to 1230°C in an oxygen- and copper oxide-containing nitrogen atmosphere which is produced by routing an oxygen-nitrogen mixture (with an oxygen-nitrogen ratio of 20:80) over copper oxide heated to 1750°C;

3. Keeping at 1230°C for 45 minutes;

4. Continue with process steps 5 - 10 as in example 1 above.

Other processes for applying copper containing materials are possible for producing the Al_2O_3-$CuAl_2O_4$ intermediate layer, for example, screen printing, coating, dipping in solutions, etc.

Other processes are also conceivable for making available copper during the AlN oxidation phase, especially physical processes, for example, sputtering of CuO at temperatures between 800 and 1300°C. The formation of an intermediate layer of $Al_2O_3CuAl_2O_4$ is also important here.

Initial substrate 1 with core 5 of AlN and dense intermediate layers 6 of Al_2O_3 and copper oxide is obtained with these aforementioned processes. The ratio of Al_2O_3 to copper oxide can be varied by the layer thickness of the previously applied copper or copper oxide in combination with the temperature and duration of firing.

Figure 2 shows initial substrate 1a which differs from the initial substrate of Figure 1 in that intermediate layers 6a there in addition to layers 7 of Al_2O_3 directly adjacent to core 5 with the portion of copper oxide have another layer 7' which is applied to each of these layers and which consists exclusively of aluminum oxide ceramic (Al_2O_3). This initial substrate 1a shown in Figure 2 can be obtained with techniques known to one skilled in the art, for example, by additional application or spraying of layers 7' in the plasma.

Figure 3 shows initial substrate 1b which differs from the initial substrate of Figure 1 in that in initial substrate 1b in intermediate layers 6b there the copper oxide is present uniformly distributed in aluminum oxide in small clusters with a diameter of less than 0.01 micron.

Figure 4 shows in cross section a substrate which is produced for example from initial substrate 1 using two copper

layers 2. Accordingly, this substrate has core 5 of AlN which ~~is~~ has provided on both sides, ~~with one~~ an intermediate layer 6, ~~at a time~~, on which ~~then on both surface sides of substrate 1c~~ a metal coating 8 is applied over a large area using the DCB process to form substrate 1c.

Figure 5 finally shows substrate 1d which differs from substrate 1c essentially in that the copper foils used to produce metal coating 8 on their surface sides have an ~~especially~~ oxygen-rich copper phase, i.e., an oxide layer with ~~especially~~ great thickness, for example, with a thickness of at least 3 microns, so that between intermediate layer 6 and each copper layer 8 there is ~~still~~ layer 9 which contains copper and copper oxide ($Cu + Cu_2O$).

The invention was described above using embodiments. It goes without saying that numerous changes or modifications are possible without departing from the inventive idea underlying the invention. Thus, using the above described technique it is especially possible to produce substrates which have a metal coating only on one side of the aluminum nitride ceramic and/or to produce these substrates which have a plurality of metal layers with interposed ceramic layers, of which at least some are then formed by the aluminum nitride ceramic provided with intermediate layers 6 or 6b.

Reference number list

1 initial substrate

2 copper foil or copper layer

3 core

4 oxide layer

5 core

6 intermediate layer

7 intermediate layer

8 metal coating

9 copper-copper oxide layer

Claims

18 A ~~1~~. Process for producing a substrate with at least one layer ~~(5)~~ of an aluminum nitride ceramic, comprising the steps of applying an ~~in which (process) one~~ auxiliary or intermediate layer ~~(6, 6a, 6b)~~ of an aluminum oxide ~~is applied~~ to at least one side of said at least one ~~this~~ layer ~~(5)~~, wherein said auxilliary ~~characterized in that~~ intermediate layer ~~(6, 6a, 6b)~~ is applied using a copper oxide-containing material such that said auxilliary or ~~the~~ intermediate layer, such that ~~at least in~~ a part of a ~~its~~ thickness of said auxilliary or intermediate layer contains ~~a portion of copper oxide of~~ about ~~roughly~~ 0.05-44 percent by weight of a copper oxide, and said at least one ~~that~~ layer ~~(5)~~ of aluminum nitride ceramic of said ~~first on~~ at least one ~~surface~~ side is provided with a layer having a thickness of about ~~roughly~~ 1.5 x 10^{-4} to 1200 x 10^{-4} micron ~~thick~~ layer of copper or copper oxide or other copper-containing compounds to form a substrate, and then treating said substrate at a temperature ranging from about ~~between roughly~~ 800 - 1300°C ~~is treated~~ in an oxygen-containing atmosphere until said ~~an~~ intermediate layer with a ~~the~~ desired thickness has formed.

19 ~~2~~. A Process according to claim 18 ~~1~~, wherein treatment in said ~~the~~ oxygen-containing atmosphere lasts until a layer thickness of about ~~roughly~~ 0.5 - 10 microns has developed for said ~~respective~~ intermediate layer ~~(6, 6a, 6b)~~.

20 ~~3~~. A Process according to claim 18 ~~1~~, wherein said aluminum nitride ceramic is treated in an oxygen-containing atmosphere, at the same time said copper-oxide containing material is reacted via the gaseous phase with said ~~the forming~~ aluminum oxide ~~(Al_2O_3)~~.

21 ~~4~~. A Process according to claim 20 ~~3~~, wherein said treatment in said ~~the~~ oxygen-containing atmosphere with a ~~portion of vaporous~~ copper-oxide containing material lasts until a layer thickness of roughly 0.5 - 10 microns has developed for said intermediate layer.

22 ~~5~~. A Process according ~~to one of~~ claim~~s~~ ~~1~~ 18, wherein after producing at least one intermediate layer, ~~(6, 6a, 6b)~~ a metal layer ~~(8)~~ is attached to ~~the latter~~ said at least one intermediate layer over its surface using an oxidized metal or copper foil and further using ~~the~~ a DCB process.

23 ~~6~~. Process according to ~~one of~~ claim~~s~~ 1, wherein said at least one layer ~~(5)~~ of aluminum ~~oxide~~ nitride ceramic is provided on both sides with ~~one~~ ~~an~~ said auxiliary or intermediate layer ~~(6, 6a, 6b) each~~, and wherein one metal or copper layer is applied to each of ~~the two~~ said intermediate layers using ~~the~~ a DCB process.

24 ~~7~~. A Substrate ~~with~~ comprising at least one layer ~~(5) essentially~~ of an of aluminum nitride ~~(AlN)~~ ceramic, ~~which is provided on at least one surface side with~~ an intermediate or auxiliary layer ~~(6, 6a, 6b)~~ which ~~contains~~ comprises aluminum oxide (Al_2O_3) and which has a thickness ~~in the range of~~ of about roughly 0.5 - 10 microns, provided on said a surface of said at least one layer of an aluminum nitride ceramic, wherein said intermediate layer ~~(6, 6a, 6b) as the additive~~ contains 0.05-44 percent by weight of at least one copper oxide, and wherein ~~the~~ a ~~proportion~~ portion of copper oxide in ~~the~~ said intermediate layer (6b) is ~~provided~~ uniformly distributed in clusters.

25 ~~8~~. A Substrate according to claim ~~7~~ 24, wherein ~~the~~ said clusters have a diameter of less than ~~roughly~~ about 0.01 microns.

~~26 9. A Substrate according to claim 24, wherein said the copper oxide in area (6) of intermediate layer (6a) adjacent to layer (5) of aluminum oxide is enriched.~~

26. ~~10.~~ A Substrate according to claim ~~7~~ 24, wherein said intermediate layer ~~(6a)~~ ~~consists of~~ comprises a first layer of aluminum oxide which is directly adjacent to ~~the~~ said layer of aluminum nitride ceramic and ~~which contains a portion of~~ a copper oxide, and ~~of adjacent~~ a second

aluminum oxide layer (7') which contains no copper ~~of~~ or copper oxide. ~~or essentially none.~~

27 ~~11.~~ A Substrate according to claim ~~7~~ 24, wherein ~~the~~ a concentration of said copper oxide in said intermediate layer ~~(6a)~~ decreases with an increasing distance from said layer ~~(5)~~ of aluminum nitride ceramic.

28 12. A Substrate according to claim ~~7~~ 24, wherein ~~the~~ said portion of copper oxide ~~is~~ present in ~~the~~ said aluminum oxide ~~as~~ is copper-aluminum-spinel ($CuAl_2O_4$ or CuAlO).

29 ~~13.~~ A Substrate according to claim ~~7~~ 24, wherein said at least one layer ~~(5)~~ of aluminum nitride is joined flat on one side via said intermediate layer ~~(6, 6a, 6b) there~~ with a copper layer or metal coating ~~(8)~~, ~~preferably with~~ a 0.1 - 0.8 mm thick ~~copper layer~~.

30 ~~14.~~ A Substrate according to claim ~~7~~ 24, wherein said copper -oxide layer ~~(8)~~ is joined via an oxygen-rich copper phase ~~(9)~~, ~~preferably via an oxygen-rich copper phase with~~ having a thickness of at least three microns, to one side of said at least on layer ~~(5)~~ of aluminum nitride ceramic or to said intermediate layer. ~~(6, 6a, 6b) there.~~

31 ~~15.~~ A Substrate according to claim ~~7~~ 24, wherein said at least one layer ~~(5)~~ of aluminum nitride contains ~~roughly~~ about 0.5 - 6 percent by weight of a binder. ~~CaO. or another binder.~~

32. A substrate according to claim 31, wherein said binder is CaO.

33 ~~16.~~ A Substrate according to claim ~~7~~ 24, wherein a ~~portion of the~~ ~~said~~ binder is ~~likewise~~ present in ~~the~~ said intermediate layer.

34. A substate ... 33. wherein said binder is CaO.

35 ~~17.~~ A Substrate according to claim ~~7~~ 24, wherein at least one copper layer ~~(8)~~ is applied to at least one side of said at least one layer ~~(5)~~ of aluminum nitride ceramic or to said intermediate layer ~~(6, 6a, 6b) there~~ in ~~the~~ a DCB process.

Abstract of the Disclosure

The invention relates to a novel ceramic substrate with at least one layer ~~essentiall~~y of aluminum nitride which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide and which has a thickness in the range of 0.5 - 10 microns, and to a process for its production.

A/ NO FEE

PROCESS FOR PRODUCING A CERAMIC SUBSTRATE AND A CERAMIC SUBSTRATE

BACKGROUND OF THE INVENTION

The invention relates to a process for producing a substrate with at least one layer of aluminum nitride ceramic, in which one auxiliary or intermediate layer of aluminum oxide is applied to at least one side of this layer.

The invention further relates to a substrate with at least one layer of aluminum nitride (AlN) which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide (Al_2O_3) and which has a thickness in the range of roughly 0.5 - 10 microns.

Ceramic substrates for electrical circuits or modules, especially for power circuits or modules, are known in the most varied versions. In particular, it is known to produce a metal coating, required for making printed conductors, terminals, etc., on an aluminum oxide ceramic using the so-called "DCB process" (direct copper bond technology) and using copper foils which are oxidized on their surfaces and which form the metallic coating. The copper oxide layer of these foils forms a eutectic with a melting point below the melting point of the copper so that by placing the foils on the ceramic and by heating all the layers they can be joined to one another, especially by melting on the copper, only in the area of the oxide layer. The DCB process is a technique known to one skilled in the art.

In power circuits, the use of an aluminum nitride ceramic instead of an aluminum oxide ceramic is desirable due to the thermal conductivity of the aluminum nitride ceramic, which is higher than that of the aluminum oxide ceramic.

In this case, the DCB process cannot be easily used for aluminum nitride ceramics.

It has been suggested to first apply a layer of aluminum oxide to the layer of aluminum nitride ceramic, such that this intermediate or auxiliary layer enables the application of a metallic coating or the copper layer using the DCB process. This intermediate layer has a roughness which improves adhesion on its exposed surface (DE-OS 35 34 886).

Furthermore, it was proposed that an aluminum oxide layer be applied to an initial substrate or carrier body of aluminum nitride, by flame spraying an aluminum oxide powder or in a screen printing process (DE-OS 38 44 264).

In all these known DCB processes, the disadvantage is that in spite of applying the auxiliary or intermediate layer of aluminum oxide, a flawless homogenous flat bond without faults is not achieved between the layer of aluminum nitride ceramic and the metal coating, but rather numerous faults occur, i.e., areas in which no bond has been formed or the metal coating has been lifted away from the ceramic by bubble formation. Thus the adhesive strength of the metal coatings and the thermal conductivity of the substrates overall are adversely affected.

To improve adhesive strength or bond quality, i.e., to reduce the bubble portion, it is proposed in DE 41 04 860.1 that oxidation of the aluminum nitride (AlN) be done to form the intermediate layer of aluminum oxide (Al_2O_3). This is accomplished in an oxygen atmosphere free of water vapor.

Conversely, it is proposed in WO 92/11 113 that the aluminum nitride be oxidized and cooled in a controlled manner under a water vapor-containing atmosphere to form the intermediate layer of aluminum oxide.

In JP 02-124 773 A (in Patent Abstracts of Japan C - 743, Vol. 14/No. 342) it is proposed that the adhesive strength of the metal coating be increased by incorporating CaO and SiO_2 into the aluminum oxide (Al_2O_3) layer, CaO and SiO_2 originating from the sintering aids of the aluminum nitride (AlN) body.

One disadvantage of the aforementioned processes, is that the process parameters must be very accurately maintained and further these processes are not applicable to highly heat-conductive aluminum nitride which contains yttrium oxide (Y_2O_3) as the sintering aid.

In JP 03-228 885 A (in Patent Abstracts of Japan C-890, Vol. 16/No. 1) it is proposed in order to increase the adhesive strength that the aluminum oxide layers be doped with one or more elements of the group Ti, V, Mo, Mb, W, Co or Ni.

In WO 92/11 113 reference is made to a publication of Kuromitsu which relates to SiO_2-Al_2O_3 intermediate layers for increasing adhesive strength.

In our own extensive studies, however, the described results of the aforementioned known processes could not be confirmed. It is thus apparent that the process parameters must be precisely controlled or negative results occur even with small deviations.

The incorporation of SiO_2 cited in WO 92/11 113 moreover also engenders the danger that SiO_2 is present not as SiO_2 bound as mullite, but as free SiO_2 which reacts at the eutectic temperature with copper oxide to form a liquid phase and consumes the Cu-Cu_2O eutectic which is necessary for the DCB process, with which the adhesive strength or bond quality would be strongly reduced.

The object of the invention is to devise a process for producing a ceramic substrate which avoids the aforementioned disadvantages and makes it possible to apply the metal coating to an aluminum oxide ceramic over a large area without faults.

SUMMARY OF THE INVENTION

To achieve this object a process for producing a substrate with at least one layer of aluminum nitride ceramic, in which an auxiliary or intermediate layer of aluminum oxide is applied to at least one side of this layer is characterized in that the intermediate layer is applied using a copper oxide-containing material such that the intermediate layer at least in part of its thickness contains a portion of copper oxide of roughly 0.05-44 percent by weight and that the layer of aluminum nitride first on at least one surface side is provided with a roughly 1.5 x 10^{-4} to 1200 x 10^{-4} micron thick layer of copper or copper oxide or other copper-containing compounds and then at a temperature between roughly 800 - 1300°C is treated in an oxygen-containing atmosphere until an intermediate layer with the desired thickness is formed.

A substrate is formed with at least one layer which comprises aluminum nitride (AlN) and which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide (Al_2O_3) and has a thickness in the range from roughly 0.5 to 10 microns, and is characterized in that the intermediate layer has an additive present from 0.05-44 percent by weight of at least one copper oxide and that the proportion of copper oxide in the intermediate layer is uniformly distributed in clusters.

In the invention the intermediate layer applied to the aluminum nitride layer contains a small portion of copper oxide which is present and bound in the form of a spinel, for example, as $CuAl_2O_4$. Surprisingly, considerable compaction of the intermediate or auxiliary layer of aluminum oxide occurs due to the portion of copper oxide, so that diffusion of the nitrogen through the intermediate layer which is responsible for the faults and bubble formation in the prior art is effectively prevented. According to one finding underlying the invention, the improved seal can be attributed to the fact that by adding copper oxide in the intermediate or auxiliary layer those temperature changes which a substrate inevitably undergoes in

conventional processes cannot lead to cracks or leaks in this intermediate layer. In this way, the passage of gas through this layer and as in the DCB process, the reaction of the aluminum nitride with the oxygen or the copper oxide of the copper foils, which normally causes the bubble fault, is effectively prevented. Compared to a pure Al_2O_3 layer, the reactivity of an Al_2O_3-$CuAl_2O_4$-intermediate layer with the liquid Cu-Cu_2O eutectic is greatly improved.

According to the finding underlying the invention the improved seal can be attributed to the reduction of pore volume and prevention of crack formation during the temperature changes which necessarily occur and which a substrate undergoes in the conventional processes as a result of the incorporation of copper oxide into the Al_2O_3 phase.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1 is a cross-sectional view of an initial substrate;

FIGURE 2 is a cross-sectional view of an initial substrate;

FIGURE 3 is a cross-sectional view of an initial substrate;

FIGURE 4 is a cross-sectional view of a substrate produced from initial substrate 1; and

FIGURE 5 is a cross-sectional view of a substrate produced from initial substrate 2, and is an alternate preferred embodiment.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The invention is detailed below using Figures 1-5 which each reproduce in a simplified representation and in cross section various possible embodiments of the substrate according to the invention.

In the figures, the initial substrate or a layer of aluminum nitride ceramic (AlN) is labelled 1.

In the embodiment assumed for Figure 1 this initial substrate 1 is to be produced, using the DCB process known to one skilled in the art, with a metal coating on each of the two sides, i.e., with a thin copper layer or foil 2, each copper foil 2 being oxidized on both surface sides, i.e., consisting of core 3 of copper (Cu) and thin copper oxide layer 4 (Cu_2O/CuO) on both surface sides. Core 3 has a much greater thickness than that of two oxide layers 4.

To be able to bond copper layers 2 using the DCB process to initial substrate 1, the latter in the embodiment shown in Figure 1 has in addition to core 5 of aluminum nitride ceramic (AlN) on both surface sides, layer 6 which consists of aluminum oxide (Al_2O_3) and which contains a portion of copper oxide (Cu_2O/CuO) in an amount between 0.05 to 44 percent by weight. The thickness of layers 6 is much less than the thickness of core 5 and is roughly between 0.5 and 10 microns.

It has been shown that only by means of the addition of copper oxide in the intermediate layer 6, there is produced a homogenous bond of initial substrate 1 with copper layers 2 even using the DCB process, i.e., a blanket bond without defects, i.e., without areas on which the bond between respective copper layer 2 and initial substrate 1 has not occurred or is disrupted by bubble formation.

As was detailed above, according to the finding underlying the invention this can only be achieved when intermediate layers 6 of aluminum oxide ceramic (Al_2O_3) which are fundamentally necessary for execution of the DCB process have the above described proportion of copper oxide, since in the conventional process steps major temperature changes of the

initial substrate cannot be avoided after applying intermediate layers 6. These temperature fluctuations, according to the finding underlying the invention, in the absence of copper oxide in intermediate layers 6, lead to cracks or leaks with the result that in the area of these leaks the aluminum nitride of core 5 reacts directly with the copper oxide of oxide layers 4, preventing the desired bond from occurring between initial substrate 1 and the copper layers, and nitrogen is released resulting in bubble formation between respective copper layer 2 and the initial substrate, according to the following reaction equation:

$$2AlN + 3Cu_2O \text{ ----- } Al_2O_3 + Cu + N_2$$

Surprisingly, the proportion of copper oxide incorporated into intermediate layers 6 causes their compaction such that these leaks or cracks do not occur for the temperature differences which are inevitable in the process in intermediate layers 6, nor do the associated disadvantages arise.

Since the thickness of intermediate layers 6 is roughly between 0.5 and 10 microns, these intermediate layers are sufficient for reliable bonding of the initial substrate with copper layers 2; the special advantage of the aluminum nitride, specifically the thermal conductivity which is much improved compared to the aluminum oxide ceramic, is however not adversely affected overall.

The initial substrate can be produced for example such that on core 5 of the aluminum nitride (AlN), there is provided on each side, a very thin layer, i.e., a layer with a thickness of roughly 1.5×10^{-4} to 1200×10^{-4} microns of copper or copper oxide or the corresponding ions of other copper-containing compounds and afterwards this core 5 is treated at a temperature in the range between roughly 800-1300°C, preferable at a temperature of 1200°C, in an oxidizing atmosphere.

The following processes are suitable, for example, for producing the initial substrate:

Example 1

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the substrate by plasma etching;

2. Application of a uniform copper layer by sputtering onto both sides of the cleaned substrate with a total amount of 1.3 mg copper at a time.

3. Heating of the substrate in an atmosphere containing N_2 and O_2 in a proportion of 20:80 to a temperature of roughly 1280°C;

4. Keeping the substrate at a temperature of 1280°C for 30 minutes;

5. Cooling to room temperature;

6. Oxidizing a copper sheet measuring 100 x 100 mm and a thickness of 0.3 mm such that a uniform copper oxide layer of roughly 0.8 microns thick results;

7. Placing the copper sheet on the AlN substrate treated in steps 1-5;

8. Heating of the bond of substrate and copper sheet to 1071°C in a gas atmosphere consisting of nitrogen with an oxygen portion of 40×10^{-6};

9. Keeping the bond at the temperature for 2.5 minutes;

10. Cooling to room temperature.

A substrate of AlN with an intermediate layer of Al_2O_3 and $CuAl_2O_4$ and a securely bonded copper layer with a peel strength greater than 50 N/mm is obtained.

Example 2:

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the aluminum substrate with ultrasound in deionized water;

2. Currentless deposition of copper with a commercial bath until 1.8 mg copper total has been deposited on each side of the substrate;

3. This is followed by process steps 3-10 of example 1.

Example 3:

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the aluminum substrate with ultrasound in deionized water;

2. Heating of the substrate in a tube furnace to 1230°C in an oxygen- and copper oxide-containing nitrogen atmosphere which is produced by routing an oxygen-nitrogen mixture (with an oxygen-nitrogen ratio of 20:80) over copper oxide heated to 1750°C;

3. Keeping at 1230°C for 45 minutes;

4. Continue with process steps 5 - 10 as in example 1 above.

Other processes for applying copper containing materials are possible for producing the Al_2O_3-$CuAl_2O_4$ intermediate layer, for example, screen printing, coating, dipping in solutions, etc.

Other processes are also conceivable for making available copper during the AlN oxidation phase, especially physical processes, for example, sputtering of CuO at temperatures between 800 and 1300°C. The formation of an intermediate layer of $Al_2O_3CuAl_2O_4$ is also important here.

Initial substrate 1 with core 5 of AlN and dense intermediate layers 6 of Al_2O_3 and copper oxide is obtained with these aforementioned processes. The ratio of Al_2O_3 to copper oxide can be varied by the layer thickness of the previously applied copper or copper oxide in combination with the temperature and duration of firing.

Figure 2 shows initial substrate 1a which differs from the initial substrate of Figure 1 in that intermediate layers 6a there in addition to layers 7 of Al_2O_3 directly adjacent to core 5 with the portion of copper oxide have another layer 7' which is applied to each of these layers and which consists exclusively of aluminum oxide ceramic (Al_2O_3). This initial substrate 1a shown in Figure 2 can be obtained with techniques known to one skilled in the art, for example, by additional application or spraying of layers 7' in the plasma.

Figure 3 shows initial substrate 1b which differs from the initial substrate of Figure 1 in that in initial substrate 1b in intermediate layers 6b there the copper oxide is present uniformly distributed in aluminum oxide in small clusters with a diameter of less than 0.01 micron.

Figure 4 shows in cross section a substrate which is produced for example from initial substrate 1 using two copper layers 2. Accordingly, this substrate has core 5 of AlN which has provided on both sides, an intermediate layer 6, on which a metal coating 8 is applied over a large area using the DCB process to form substrate 1c.

Figure 5 finally shows substrate 1d which differs from substrate 1c essentially in that the copper foils used to produce metal coating 8 on their surface sides have an oxygen-rich copper phase, i.e., an oxide layer with great thickness, for example, with a thickness of at least 3 microns, so that between intermediate layer 6 and each copper layer 8 there is layer 9 which contains copper and copper oxide ($Cu + Cu_2O$).

The invention was described above using embodiments. It goes without saying that numerous changes or modifications are possible without departing from the inventive idea underlying the invention. Thus, using the above described technique it is

especially possible to produce substrates which have a metal coating only on one side of the aluminum nitride ceramic and/or to produce these substrates which have a plurality of metal layers with interposed ceramic layers, of which at least some are then formed by the aluminum nitride ceramic provided with intermediate layers 6 or 6b.

Reference number list

1	initial substrate
2	copper foil or copper layer
3	core
4	oxide layer
5	core
6	intermediate layer
7	intermediate layer
8	metal coating
9	copper-copper oxide layer

WHAT IS CLAIMED IS:

18. A process for producing a substrate with at least one layer of an aluminum nitrate ceramic, comprising the steps of applying an auxiliary or intermediate layer of an aluminum oxide to at least one side of said at least one layer, wherein said auxiliary intermediate layer is applied using a copper oxide-containing material such that said auxiliary or intermediate layer, such that a part of a thickness of said auxiliary or intermediate layer contains about 0.05-44 percent by weight of a copper oxide and said at least one layer of aluminum nitride ceramic of said at least one side is provided with a layer of copper or copper oxide or other copper-containing compounds having a thickness of about 1.5 x 10^{-4} micron thick to form a substrate; and then treating said substrate at a temperature ranging from about 800 - 1300°C is in an oxygen-containing atmosphere until said intermediate layer with a desired thickness has formed.

19. A process according to claim 18, wherein treatment in said oxygen-containing atmosphere lasts until a layer thickness of about 0.5 - 10 microns has developed for said intermediate layer.

20. A process according to claim 18, wherein said aluminum nitride ceramic is treated in an oxygen-containing atmosphere, at the same time said copper-oxide containing material is reacted via the gaseous phase with said aluminum oxide.

21. A process according to claim 20, wherein said treatment in said oxygen-containing atmosphere with a copper-oxide-containing material lasts until a layer thickness of roughly 0.5 - 10 microns has developed for said intermediate layer.

22. A process according to claim 18, wherein after producing at least one intermediate layer, a metal layer is attached to said at least one intermediate layer over its surface using an oxidized metal or copper foil and further using a DCB process.

23. A process according to claim 18, wherein said at least one layer of aluminum nitride ceramic is provided on both sides with said auxiliary or intermediate layer, and wherein one metal or copper layer is applied to each of said intermediate layers using a DCB process.

24. A substrate comprising at least one layer of an aluminum nitride ceramic, an intermediate or auxiliary layer which comprises aluminum oxide (Al_2O_3) and which has a thickness of about roughly 0.5 - 10 microns provided on said surface of said at least one layer of an aluminum nitride ceramic, wherein said intermediate layer contains 0.05-44 percent by weight of at least one copper oxide, and wherein a portion of copper oxide in said intermediate layer is uniformly distributed in clusters.

25. A substrate according to claim 24, wherein said clusters have a diameter of less than about 0.01 microns.

26. A substrate according to claim 24, wherein said intermediate layer comprises a first layer of aluminum oxide which is directly adjacent to said layer of aluminum nitride ceramic and a copper oxide, and a second aluminum oxide layer which contains no copper or copper oxide.

27. A substrate according to claim 24, wherein a concentration of said copper oxide in said intermediate layer decreases with an increasing distance from said layer of aluminum nitride ceramic.

28. A substrate according to claim 24, wherein said portion of copper oxide present in said aluminum oxide is copper-aluminum-spinel ($CuAl_2O_4$ or CuAlO).

29. A substrate according to claim 24, wherein said at least one layer of aluminum nitride is joined flat on one side via said intermediate layer with a 0.1 - 0.8 mm thick copper layer or metal coating.

30. A substrate according to claim 24, wherein said copper-oxide layer is joined via an oxygen-rich copper phase, having a thickness of at least three microns, to one side of said at least on layer of aluminum nitride ceramic or to said intermediate layer.

31. A substrate according to claim 24, wherein said at least one layer of aluminum nitride contains about 0.5 - 6 percent by weight of a binder.

32. A substrate according to claim 31, wherein said binder is CaO.

33. A substrate according to claim 24, wherein a binder is present in said intermediate layer.

34. A substrate according to claim 33, wherein said binder is CaO.

35. A substrate according to claim 24, wherein at least one copper layer is applied to at least one side of said at least one layer of aluminum nitride ceramic or to said intermediate layer in a DCB process.

ABSTRACT OF THE DISCLOSURE

The invention relates to a novel ceramic substrate with at least one layer of aluminum nitride which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide and which has a thickness in the range of 0.5 - 10 microns, and to a process for its production.

VERIFICATION OF TRANSLATION

I, Joseph Crabbs, a translator with Chillson Translating Service, 3326 Brantly Road, Glenwood, Maryland 21738, hereby declare as follows:

That I am familiar with the German and English languages;

That I am capable of translating from German to English;

That the translation attached hereto is a true and accurate translation of the German application titled, "Process for producing a ceramic substrate and a ceramic substrate;"

That all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true;

And further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any registration resulting therefrom.

By Joseph W Crabbs

Executed this 30 day of January 1997.

Process for producing a ceramic substrate and a ceramic substrate

The invention relates to a process for producing a substrate with at least one layer of aluminum nitride ceramic, in which (process) one auxiliary or intermediate layer of aluminum oxide is applied to at least one side of this layer.

The invention relates furthermore to a substrate with at least one layer essentially of aluminum nitride (AlN) which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide (Al_2O_3) and which has a thickness in the range of roughly 0.5 - 10 microns.

Ceramic substrates for electrical circuits or modules, especially for power circuits or modules, are known in the most varied versions. In particular, producing the metal coating required for making printed conductors, terminals, etc. on an aluminum oxide ceramic using the so-called "DCB process" (direct copper bond technology) and using copper foils which are oxidized on their surfaces and which form the metallic coating is known. The copper oxide layer of these foils forms a eutectic with a melting point below the melting point of the copper so that by placing the foils on the ceramic and by heating all layers they can be joined to one another, especially by melting on the copper essentially only in the area of the oxide layer. The DCB process is a technique known to one skilled in the art.

Especially in power circuits is the use of an aluminum nitride ceramic instead of an aluminum oxide ceramic desirable

due to the thermal conductivity of the aluminum nitride ceramic which is higher than that of the aluminum oxide ceramic.

But here the disadvantage is essentially that the conventional and proven DCB process cannot be easily used in aluminum nitride ceramics.

It has therefore be suggested that first a layer of aluminum oxide be applied to the layer of aluminum nitride ceramic, such that this intermediate or auxiliary layer which is designed to enable application of a metallic coating or the copper layer using the DCB process has a roughness which improves adhesion on its exposed surface (DE-OS 35 34 886).

Furthermore, it was proposed that an aluminum oxide layer be applied to an initial substrate or carrier body of aluminum nitride, especially by flame spraying an aluminum oxide powder or in a screen printing process (DE-OS 38 44 264).

In all these known processes the disadvantage is that in spite of applying the auxiliary or intermediate layer of aluminum oxide, a flawless homogenous flat bond without faults between the layer of aluminum nitride ceramic and the metal coating is not achieved in the DCB process, but rather numerous faults occur, i.e., areas in which no bond has been formed or the metal coating has been lifted away from the ceramic by bubble formation. Thus the adhesive strength of the metal coatings and the thermal conductivity of the substrates overall are adversely affected.

To improve adhesive strength or bond quality, i.e., to reduce the bubble portion, it is proposed in DE 41 04 860.1 that oxidation of the aluminum nitride (AlN) be done to form the

intermediate layer of aluminum oxide (Al_2O_3) in an oxygen atmosphere free of water vapor.

Conversely, it is proposed in WO 92/11 113 that the aluminum nitride be oxidized and cooled in a controlled manner under a water vapor-containing atmosphere to form the intermediate layer of aluminum oxide.

In JP 02-124 773 A (in Patent Abstracts of Japan C - 743, Vol. 14/No. 342) it is proposed that the adhesive strength of the metal coating be increased by incorporating CaO and SiO_2 into the aluminum oxide (Al_2O_3) layer, CaO and SiO_2 originating from the sintering aids of the aluminum nitride (AlN) body.

One disadvantage of the aforementioned known process is among others that the process parameters must be very accurately maintained and these processes not be applied especially to highly heat-conductive aluminum nitride which contains yttrium oxide (Y_2O_3) as the sintering aid.

In JP 03-228 885 A (in Patent Abstracts of Japan C-890, Vol. 16/No. 1) it is proposed in order to increase the adhesive strength that the aluminum oxide layers be doped with one or more elements of the group Ti, V, Mo, Mb, W, Co or Ni.

In WO 92/11 113 reference is made to a publication of Kuromitsu which relates to SiO_2-Al_2O_3 intermediate layers for increasing adhesive strength.

In our own extensive studies however the described results of the aforementioned known processes could not be confirmed. It is thus apparent that the process parameters must be precisely

controlled such that negative results occur even with small deviations.

The incorporation of SiO_2 cited in WO 92/11 113 moreover also engenders the danger that SiO_2 is present not as SiO_2 bound as mullite, but as free SiO_2 which reacts at the eutectic temperature with copper oxide to form a liquid phase and consumes the Cu-Cu_2O eutectic which is necessary for the DCB process, with which the adhesive strength or bond quality would be strongly reduced.

The object of the invention is to devise a process for producing a ceramic substrate which avoids the aforementioned disadvantages and makes it possible to apply the metal coating to an aluminum oxide ceramic over a large area without faults.

To achieve this object a process for producing a substrate with at least one layer of aluminum nitride ceramic, in which (process) an auxiliary or intermediate layer of aluminum oxide is applied to at least one side of this layer is characterized in that the intermediate layer is applied using a copper oxide-containing material such that the intermediate layer at least in part of its thickness contains a portion of copper oxide of roughly 0.05-44 percent by weight and that the layer of aluminum nitride first on at least one surface side is provided with a roughly 1.5×10^{-4} to 1200×10^{-4} micron thick layer of copper or copper oxide or other copper-containing compounds and then at a temperature between roughly 800 - 1300°C is treated in an oxygen-containing atmosphere until an intermediate layer with the desired thickness as formed.

A substrate with at least one layer which consists essentially of aluminum nitride (AlN) and which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide (Al_2O_3) and has a thickness in the range from roughly 0.5 to 10 microns is characterized in that the intermediate layer as the additive contains 0.05-44 percent by weight of at least one copper oxide and that the proportion of copper oxide in the intermediate layer is provided uniformly distributed in clusters.

In the invention the intermediate layer applied to the aluminum nitride layer contains a small portion of copper oxide which is present bound in the form of a spinel, for example, as $CuAl_2O_4$. Surprisingly, considerable compaction of the intermediate or auxiliary layer of aluminum oxide occurs due to the portion of copper oxide, so that diffusion of the nitrogen through the intermediate layer which is responsible for the faults and bubble formation in the prior art is effectively prevented. According to one finding underlying the invention the improved seal can be attributed to the fact that by adding copper oxide in the intermediate or auxiliary layer those temperature changes which a substrate inevitably undergoes in the conventional processes cannot lead to cracks or similar leaks in this intermediate layer. In this way passage of gas through this layer and thus especially in the DCB process also the reaction of the aluminum nitride with the oxygen or the copper oxide of the copper foils used in the DCB process which causes the bubble fault are effectively prevented. Compared to the pure Al_2O_3

layer in addition in the invention the reactivity of the Al_2O_3-$CuAl_2O_4$-intermediate layer with the liquid Cu-Cu_2O eutectic is greatly improved during the DCB process.

According to the finding underlying the invention the improved seal can be attributed to the reduction of pore volume and prevention of crack formation during the temperature changes which necessarily occur and which a substrate undergoes in the conventional processes as a result of the incorporation of copper oxide into the Al_2O_3 phase.

Developments of the invention are the subject of the subclaims.

The invention is detailed below using Figures 1-5 which each reproduce in a simplified representation and in cross section various possible embodiments of the substrate according to the invention.

In the figures the initial substrate or a layer of aluminum nitride ceramic (AlN) is labelled 1.

In the embodiment assumed for Figure 1 this initial substrate 1 is to be provided, using the DCB process known to one skilled in the art, with a metal coating on each of the two sides, i.e., with thin copper layer or foil 2, each copper foil 2 being oxidized on both surface sides, i.e., consisting of core 3 of copper (Cu) and thin copper oxide layer 4 (Cu_2O/CuO) on both surface sides. Core 3 has a much greater thickness than that of two oxide layers 4.

To be able to bond copper layers 2 using the DCB process to initial substrate 1, the latter in the embodiment shown in Figure

1 has in addition to core 5 of aluminum nitride ceramic (AlN) on both surface sides layer 6 which consists of aluminum oxide (Al_2O_3) and which contains a portion of copper oxide (Cu_2O/CuO) in an amount between 0.05 to 44 percent by weight. The thickness of layers 6 is much less than the thickness of core 5 and is roughly between 0.5 and 10 microns.

It has been shown that only by means of the portion of copper oxide in intermediate layer 6 is a homogenous bond of initial substrate 1 with copper layers 2 possible using the DCB process, i.e., especially a blanket bond without defects, i.e., without areas on which the bond between respective copper layer 2 and initial substrate 1 has not occurred or is disrupted by bubble formation.

As was detailed above, according to the finding underlying the invention this can only be achieved when intermediate layers 6 of aluminum oxide ceramic (Al_2O_3) which are fundamentally necessary for execution of the DCB process have the above described proportion of copper oxide, since in the conventional process steps major temperature changes of the initial substrate cannot be avoided in particular after applying intermediate layers 6 and during the DCB process. These temperature fluctuations according to the finding underlying the invention in the absence of copper oxide in intermediate layers 6 lead there to cracks or leaks with the result that in the area of these leaks the aluminum nitride of core 5 reacts directly with the copper oxide of oxide layers 4 without the desired bond occurring between initial substrate 1 and the copper layers, rather

nitrogen is released with the result of bubble formation between respective copper layer 2 and the initial substrate according to the following reaction equation:

$2AlN + 3Cu_2O$ ----- $Al_2O_3 + Cu + N_2$

Surprisingly, the proportion of copper oxide incorporated into intermediate layers 6 causes their compaction such that these leaks or cracks do not occur for the temperature differences which are inevitable in the process in intermediate layers 6, nor do the associated disadvantages arise.

Since the thickness of intermediate layers 6 is roughly between 0.5 and 10 microns, these intermediate layers are sufficient for reliable bonding of the initial substrate with copper layers 2; the special advantage of the aluminum nitride, specifically the thermal conductivity which is much improved compared to the aluminum oxide ceramic, is however not adversely affected overall.

The initial substrate can be produced for example such that on core 5 of the aluminum nitride (AlN) on each side a very thin layer, i.e., a layer with a thickness of roughly 1.5×10^{-4} to 12000×10^{-4} microns of copper or copper oxide or the corresponding ions of other copper-containing compounds is applied and afterwards this core 5 is treated at a temperature in the range between roughly 800-1300°C, preferable at a temperature of 1200°C, in an oxidizing atmosphere.

The following processes are suitable for example for producing the initial substrate:

Example 1

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the substrate by plasma etching;

2. Application of a uniform copper layer by sputtering onto both sides of the cleaned substrate with a total amount of 1.3 mg copper at a time.

3. Heating of the substrate in an atmosphere containing N_2 and O_2 in a proportion of 20:80 to a temperature of roughly 1280°C;

4. Keeping the substrate at a temperature of 1280°C for 30 minutes;

5. Cooling to room temperature;

6. Oxidizing a copper sheet measuring 100 x 100 mm and a thickness of 0.3 mm such that a uniform copper oxide layer of roughly 0.8 microns thick results;

7. Placing the copper sheet on the AlN substrate treated in steps 1-5;

8. Heating of the bond of substrate and copper sheet to 1071°C in a gas atmosphere consisting of nitrogen with an oxygen portion of 40×10^{-6};

9. Keeping the bond at the temperature for 2.5 minutes;

10. Cooling to room temperature.

A substrate of AlN with an intermediate layer of Al_2O_3 and $CuAl_2O_4$ and a securely bonded copper layer with a peel strength greater than 50 N/mm is obtained.

Example 2:

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the aluminum substrate with ultrasound in deionized water;

2. Currentless deposition of copper with a commercial bath until 1.8 mg copper total has been deposited on each side of the substrate;

3. This is followed by process steps 3-10 of example 1.

Example 3:

An AlN substrate consisting of roughly 96% AlN and with roughly 4% Y_2O_3 with a thickness of 0.63 mm and with dimensions of 108 x 108 mm is subjected to the following process steps:

1. Cleaning of the aluminum substrate with ultrasound in deionized water;

2. Heating of the substrate in a tube furnace to 1230°C in an oxygen- and copper oxide-containing nitrogen atmosphere which is produced by routing an oxygen-nitrogen mixture (with an oxygen-nitrogen ratio of 20:80) over copper oxide heated to 1750°C;

3. Keeping at 1230°C for 45 minutes;

4. Continue with process steps 5 - 10 as in example 1 above.

Other processes for applying copper containing materials are possible for producing the Al_2O_3-$CuAl_2O_4$ intermediate layer, for example, screen printing, coating, dipping in solutions, etc.

Other processes are also conceivable for making available copper during the AlN oxidation phase, especially physical processes, for example, sputtering of CuO at temperatures between 800 and 1300°C. The formation of an intermediate layer of $Al_2O_3CuAl_2O_4$ is also important here.

Initial substrate 1 with core 5 of AlN and dense intermediate layers 6 of Al_2O_3 and copper oxide is obtained with these aforementioned processes. The ratio of Al_2O_3 to copper oxide can be varied by the layer thickness of the previously applied copper or copper oxide in combination with the temperature and duration of firing.

Figure 2 shows initial substrate 1a which differs from the initial substrate of Figure 1 in that intermediate layers 6a there in addition to layers 7 of Al_2O_3 directly adjacent to core 5 with the portion of copper oxide have another layer 7′ which is applied to each of these layers and which consists exclusively of aluminum oxide ceramic (Al_2O_3). This initial substrate 1a shown in Figure 2 can be obtained with techniques known to one skilled in the art, for example, by additional application or spraying of layers 7′ in the plasma.

Figure 3 shows initial substrate 1b which differs from the initial substrate of Figure 1 in that in initial substrate 1b in intermediate layers 6b there the copper oxide is present uniformly distributed in aluminum oxide in small clusters with a diameter of less than 0.01 micron.

Figure 4 shows in cross section a substrate which is produced for example from initial substrate 1 using two copper

layers 2. Accordingly, this substrate has core 5 of AlN which is provided on both sides with one intermediate layer 6 at a time, on which then on both surface sides of substrate 1c metal coating 8 is applied over a large area using the DCB process.

Figure 5 finally shows substrate 1d which differs from substrate 1c essentially in that the copper foils used to produce metal coating 8 on their surface sides have an especially oxygen-rich copper phase, i.e., an oxide layer with especially great thickness, for example, with a thickness of at least 3 microns, so that between intermediate layer 6 and each copper layer 8 there is still layer 9 which contains copper and copper oxide (Cu + Cu_2O).

The invention was described above using embodiments. It goes without saying that numerous changes or modifications are possible without departing from the inventive idea underlying the invention. Thus, using the above described technique it is especially possible to produce substrates which have a metal coating only on one side of the aluminum nitride ceramic and/or to produce these substrates which have a plurality of metal layers with interposed ceramic layers, of which at least some are then formed by the aluminum nitride ceramic provided with intermediate layers 6 or 6b.

Reference number list

1 initial substrate

2 copper foil or copper layer

3 core

4 oxide layer

5 core

6 intermediate layer

7 intermediate layer

8 metal coating

9 copper-copper oxide layer

Claims

1. Process for producing a substrate with at least one layer (5) of aluminum nitride ceramic, in which (process) one auxiliary or intermediate layer (6, 6a, 6b) of aluminum oxide is applied to at least one side of this layer (5), characterized in that intermediate layer (6, 6a, 6b) is applied using a copper oxide-containing material such that the intermediate layer at least in part of its thickness contains a portion of copper oxide of roughly 0.05-44 percent by weight, and that layer (5) of aluminum nitride first on at least one surface side is provided with a roughly 1.5×10^{-4} to 1200×10^{-4} micron thick layer of copper or copper oxide or other copper-containing compounds and then at a temperature between roughly 800 - 1300°C is treated in an oxygen-containing atmosphere until an intermediate layer with the desired thickness has formed.

2. Process according to claim 1, wherein treatment in the oxygen-containing atmosphere lasts until a layer thickness of roughly 0.5 - 10 microns has developed for respective intermediate layer (6, 6a, 6b).

3. Process according to claim 1, wherein aluminum nitride is treated in an oxygen-containing atmosphere, at the same time copper-oxide containing material is reacted via the gaseous phase with the forming aluminum oxide (Al_2O_3).

4. Process according to claim 3, wherein treatment in the oxygen-containing atmosphere with a portion of vaporous copper oxide lasts until a layer thickness of roughly 0.5 - 10 microns has developed.

5. Process according to one of claims 1, wherein after producing at least one intermediate layer (6, 6a, 6b) metal layer (8) is attached to the latter over its surface using a oxidized metal or copper foil and using the DCB process.

6. Process according to one of claims 1, wherein at least one layer (5) of aluminum oxide ceramic is provided on both sides with one intermediate layer (6, 6a, 6b) each, and wherein one metal or copper layer is applied to each of the two intermediate layers using the DCB process.

7. Substrate with at least one layer (5) essentially of aluminum nitride (AlN) which is provided on at least one surface side with intermediate or auxiliary layer (6, 6a, 6b) which contains aluminum oxide (Al_2O_3) and which has a thickness in the range of roughly 0.5 - 10 microns, wherein intermediate layer (6, 6a, 6b) as the additive contains 0.05-44 percent by weight of at least one copper oxide, and wherein the proportion of copper oxide in ~~the~~ intermediate layer (6b) is provided uniformly distributed in clusters.

8. Substrate according to claim 7, wherein the clusters have a diameter of less than roughly 0.01 microns.

9. Substrate according to claim 7, wherein the copper oxide in area (6) of intermediate layer (6a) adjacent to layer (5) of aluminum oxide is enriched.

10. Substrate according to claim 7, wherein intermediate layer (6a) consists of a first layer of aluminum oxide which is directly adjacent to the layer of aluminum nitride and which contains a portion of copper oxide, and of adjacent second

aluminum oxide layer (7') which contains no copper of copper oxide or essentially none.

11. Substrate according to claim 7, wherein the concentration of copper oxide in intermediate layer (6a) decreases with increasing distance from layer (5) of aluminum nitride.

12. Substrate according to claim 7, wherein the portion of copper oxide is present in the aluminum oxide as copper-aluminum-spinel ($CuAl_2O_4$ or CuAlO).

13. Substrate according to claim 7, wherein layer (5) of aluminum nitride is joined flat on one side via intermediate layer (6, 6a, 6b) there with a copper layer or metal coating (8), preferably with a 0.1 - 0.8 mm thick copper layer.

14. Substrate according to claim 7, wherein copper layer (8) is joined via oxygen-rich copper phase (9), preferably via an oxygen-rich copper phase with a thickness of at least three microns, to one side of layer (5) of aluminum nitride or to intermediate layer (6, 6a, 6b) there.

15. Substrate according to claim 7, wherein at least one layer (5) of aluminum nitride contains roughly 0.5 - 6 percent by weight CaO or another binder.

16. Substrate according to claim 7, wherein a portion of the binder is likewise present in the intermediate layer.

17. Substrate according to claim 7, wherein at least one copper layer (8) is applied to at least one side of at least one layer (5) of aluminum nitride or to intermediate layer (6, 6a, 6b) there in the DCB process.

Abstract

The invention relates to a novel ceramic substrate with at least one layer essentially of aluminum nitride which is provided on at least one surface side with an intermediate or auxiliary layer which contains aluminum oxide and which has a thickness in the range of 0.5 - 10 microns, and to a process for its production.

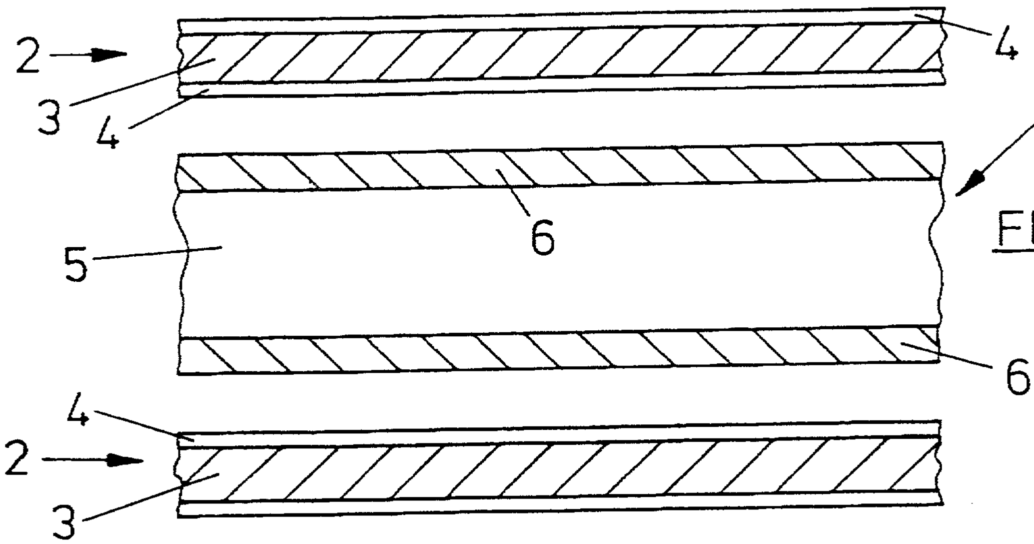

FIG.1

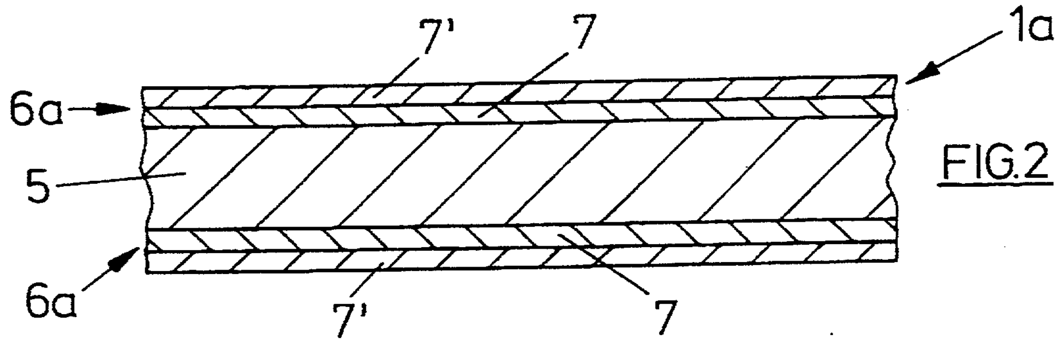

FIG.2

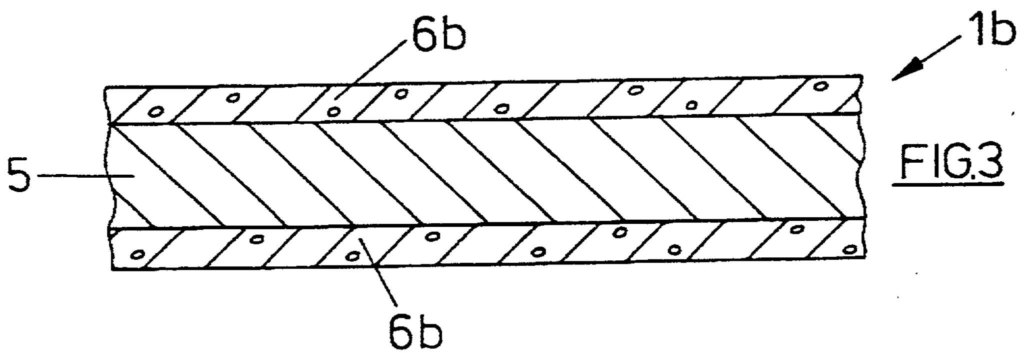

FIG.3

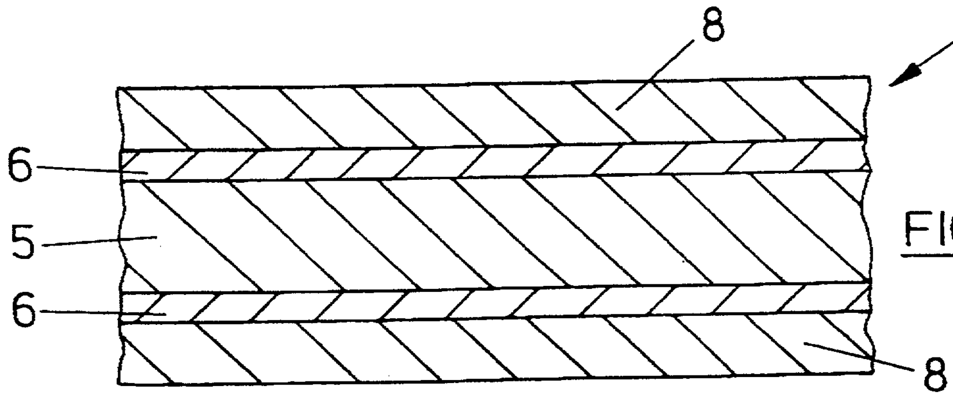

FIG.4

FIG.5